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DOE/NASA/0176-80/3
CR 165179

PREPARATION AND EVALUATION OF ADVANCED ELECTROCATALYSTS FOR PHOSPHORIC
ACID FUEL CELLS

3RD QUARTERLY REPORT
(NASA-CR-165179) PREPARATION AND EVALUATION
OF ADVANCED ELECTROCATALYSTS FOR PHOSPHORIC
ACID FUEL CELLS Quarterly Report, Jul. -
Sep. 1980 (Stonehart Associates, Inc.,
Madison, Conn.) 30 p HC A03/MF A01 CSCL 10A G3/44

N81-17527

Unclas
41405

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July - September 1980

PREPARED FOR:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Lewis Research Center
Under Contract DEN-3-176

FOR:

U.S. DEPARTMENT OF ENERGY
Energy Technology
Division of Fossil Fuel Utilization



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1. Report No. NASA CR 165179		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle PREPARATION AND EVALUATION OF ADVANCED ELECTRO-CATALYSTS FOR PHOSPHORIC ACID FUEL CELLS				5. Report Date September 1980	
				6. Performing Organization Code	
7. Author(s) Dr. Paul Stonehart John Baris and Peter Pagliaro				8. Performing Organization Report No.	
				10. Work Unit No.	
9. Performing Organization Name and Address Stonehart Associates, Inc. 17 Cottage Road Madison, Ct. 06443				11. Contract or Grant No. DEF 3-176	
				13. Type of Report and Period Covered Contractor Report	
12. Sponsoring Agency Name and Address U. S. Department of Energy Energy Technology Division of Fossil Fuel Utilization				14. Sponsoring Agency Code DOE/NASA0176-80/3	
15. Supplementary Notes 3rd Quarterly Report Mail Stop 49-5, NASA-Lewis Research Center, Project Manager: Dr. M. Lauver, 21000 Brookpark Road, Cleveland, Ohio 44135					
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17. Key Words (Suggested by Author(s)) fuel cells; electrocatalysts; electro-chemical power generation; phosphoric acid			18. Distribution Statement Unclassified-unlimited DOE Category UC-404 STAR Category 44		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages 27	
				22. Price*	

* For sale by the National Technical Information Service, Springfield, Virginia 22161

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ABSTRACT

This report encompasses the third three month period on contract NASA/DEN 3-176. In this report results are presented for hydrogen oxidation and hydrogen oxidation poisoned by carbon monoxide at levels between 0 and 30%. Due to the high activities that are now being observed for our platinum based electrocatalysts, the hydrogen concentrations have been reduced to 10% levels in the gas supplies. This procedure allows a better evaluation of the extended performances for these catalysts. Perturbation techniques have been used to determine that a mechanism for the efficient operation of our porous gas diffusion electrodes is diffusion of the carbon monoxide out of the electrode structure through the electrolyte film on the electrocatalyst. A survey of the literature on platinum group materials (PGM) has been carried out so that an identification of successful electrocatalysts can be made. Two PGM electrocatalysts were prepared and performance data for hydrogen oxidation in hot phosphoric acid in the presence of high carbon monoxide concentrations showed that they matched the best platinum on carbon electrocatalysts but with an electrocatalyst cost that was half of the platinum catalyst cost. Further improvements are now obvious, so that the anode electrocatalyst costs can be further reduced.

1.

OBJECTIVE AND SCOPE OF WORK

The overall objective of this electrocatalysis program is to define the feasibility of lowering the electrocatalyst cost and increasing the activity in phosphoric acid fuel cells, as a way to increase the commercial viability of fuel cells for producing electric power.

The specific objectives of the present tasks are the preparation of a series of high surface area electrocatalysts for evaluation in phosphoric acid fuel cells. This involves fabrication of efficient gas-diffusion electrode structures and determining their electrochemical parameters for hydrogen oxidation and oxygen reduction. When possible, new experimental techniques and theoretical interpretations will be forwarded towards an understanding of the relevant electrochemical parameters.

2. Summary of Previous Work

This report covers the third three month period on NASA DEN 3-176.

The first quarterly report showed the results for hydrogen oxidation and hydrogen oxidation poisoned by carbon monoxide at low levels (1% and 10%), for platinum supported on a furnace black (Vulcan XC-72R). These electrocatalysts had been prepared using impregnation techniques for catalyzing the carbon support. In addition, flooded electrode structures were designed in order to identify the Tafel slope for the oxygen reduction reaction in phosphoric acid over a temperature range of 120°C - 210°C. From this preliminary work a better understanding of the electrocatalyst activities under fuel cell conditions was obtained. In addition, the first studies of the effects of the electrode structures were begun so that optimized Teflon-bonded gas-diffusion electrode structures can be prepared.

In the second three month period, results were presented for hydrogen oxidation, and hydrogen oxidation poisoned by carbon monoxide at levels between 0 and 30% in the fuel gas stream for platinum supported on Vulcan XC-72R with optimized gas-diffusion electrode structures (50% PTFE), and platinum supported on Consel I (steam-treated acetylene black) with flooded electrode structures (2½% PTFE) and semi-optimized gas-diffusion electrodes (30% PTFE). The electrode performances were dependent on the thicknesses of the electrolyte films within the porous electrode structures so that the electrode performances were limited to diffusion of the carbon monoxide out of the electrode structures. Apparent carbon monoxide adsorption isotherms and the available platinum electrocatalyst surface areas have been derived. This has provided a new interpretation for the operation of fuel cell anode electrocatalyst structures under real operating conditions. In addition, a new interpretation for the activity of highly dispersed platinum crystallites on carbon for oxygen reduction was proposed. The specific activities for oxygen reduction on platinum on carbon electrocatalysts appear to be related to the platinum inter-crystallite distances on the carbon surfaces.

3. Technical Progress

3.2. Task 2 - Anodic Activities of Platinum-Carbon Electrocatalysts

Previously, in the report issued for the last quarter, we had shown how the carbon monoxide isotherms on platinum were sensitive to temperature and electrode potential. Due to the improving electrode structures, as we gain experience with working with the different electrocatalysts, we had lowered the hydrogen concentration in the fuel gas stream to 10% levels while maintaining the carbon monoxide poison levels up to 30%. Based on the changes in the apparent available platinum surface area, $(1-\theta_{CO})$, for flooded and unflooded electrode structures (see Figure 10, this contract quarterly report #2, April-June 1980), we had come to the conclusion that a critical feature determining the performance for anodes was not so much the true adsorption isotherm for carbon monoxide on the platinum but diffusion of the carbon monoxide out of the electrode structure after the hydrogen had been oxidized. It is easy to see that the carbon monoxide levels within the porous electrode structure must increase as the hydrogen in the gas stream is electrochemically oxidized. This will set up a concentration gradient between the carbon monoxide concentration within the porous structure and carbon monoxide concentration in the bulk gas phase.

In order to further resolve the operation of porous electrodes in the presence of carbon monoxide/hydrogen mixtures, we determined the polarization characteristics of a series of electrode structures containing different PTFE levels. It was our thinking that we would try to produce a smooth transition from a fully flooded electrode to a highly efficient gas diffusion electrode and measure the characteristic polarizations as a function of PTFE level. The polarization data are shown in Figures 1-4 using 2½%, 10%, 20%, and 30% PTFE respectively. In all instances the electrocatalyst was 10% platinum on steam-treated acetylene black (Consel I). Electrode performances were obtained on pure hydrogen, 10% hydrogen, and 10% hydrogen containing 1%, 2%, 10% and 30% carbon monoxide.

The data shown in Figures 1-4 show an abrupt transition in performance with PTFE level between 10% and 20%. It is possible that there are other parameters for forming the electrode structures that we do not have under sufficient control.

In order to identify the formation and relaxation of the carbon monoxide concentration gradient within the porous electrodes, the electrode structures shown in Figures 9-12 were subjected to a series of perturbation experiments. In these experiments the electrodes were maintained under the different gas environments and switched both potentiostatically or galvanostatically from the open circuit values to some characteristic operating value. After maintaining this operating characteristic for a short period of time the electrode was restored to the open circuit condition. The current and potential transients were recorded digitally using a transient analyzer. This was displayed on an oscilloscope and recorded on our XY recorder. By recording the information digitally we were able to analyze the transients point by point.

The actual relaxation curves for the transients are shown in Figures 5-8. These curves were reproduced onto the paper directly from the XY recorder and have been scaled manually. The experimental conditions are defined in the legends of the figures. Analyses of the relaxation curves are given in Table I.

Table I. Electrodes at 180°C 10% Pt on Consel I
Nominal Anode Gas Environment 2% CO; 10% H₂; 88% N₂

<u>PTFE Level</u>	<u>Current Pulse</u>	<u>Half time to Max. Potential</u>	<u>Half time to Relax to Open Circuit</u>
2.5%	100 mA	5.8 sec.	0.78 sec.
10.0%	100	2.0	0.84
20.0%	1000	0.05	0.015
30.0%	1000	0.008	0.007

Here we have chosen to show the half time to reach the maximum potential and the half time after removing the galvanostatic transient to relax back to open circuit conditions. Due to the very great differences in performances between the 2½% and 10% on the one hand and the 20% and 30% PTFE levels on the other hand, two current densities were used. For the former electrodes a 100 mA perturbation was used and in the latter cases a 1000 mA perturbation was used. The relaxations given in Table I are for gas stream containing nominally 10% hydrogen, 2% carbon monoxide and 88% nitrogen. Other relaxation curves were obtained at different carbon monoxide levels but they are not shown in this report. Table I clearly shows the trend in relaxation times that we had expected intuitively. What is surprising is the very large differences in relaxation times

between the flooded electrode structures (2½% PTFE) and the electrode containing 30% PTFE. There is a thousand times difference in the characteristic half times which suggests that measurements of this sort are indeed very sensitive to the electrode structures and electrolyte films on the electrocatalysts within the electrode structures. Following the galvanostatic relaxation curves, a series of potentiostatic transients were also obtained on these electrodes in order to provide further information regarding the carbon monoxide diffusion out of the electrode structure as the limiting factor in controlling the performance of the porous gas diffusion electrodes.

Figures 9, 10, 11 and 12 show the responses of electrodes containing 2½%, 10%, 20% and 30% PTFE with a potential step. The potential step was greater for the 2½% and 10% PTFE electrodes since the response times were slower than the response times for the 20% and 30% PTFE electrodes. The potentiostatic transient causes an instantaneous charging of the double-layer which then decays due to the transmission line through the electrode structure. This response is shown as the upper line in Figures 9-12. The half times for relaxation back to open circuit are given in Table II. Only the sets of data for a nominal gas anode stream of 2% carbon monoxide, 10% hydrogen and 80% nitrogen are shown. Other relaxation curves were obtained at different carbon monoxide levels but they are not shown in this report.

Table II. Electrodes at 180°C 10% Pt on Consel I
Nominal Anode Gas Environment 2% CO; 10% H₂; 88% N₂

<u>PTFE level</u>	<u>Potential step height</u>	<u>Half-time to relax to open-circuit</u>
2.5%	650 mV	0.8 sec.
10.0%	700	0.7
20.0%	200	0.006
30.0%	380	0.004

3.4. Task 4. Electrocatalyst Literature Survey and Selection

The purpose of this task is to review the technical and patent literature to identify platinum based materials that will be suitable for use as phosphoric acid fuel cell catalysts. In particular, the chosen materials must be capable of being prepared in a high surface area form and to be resistant to corrosion. With regard to the cathode, promising materials should have more activity for oxygen reduction than the activities so far exhibited for platinum. For the anode, the activities should be more active than platinum when operating on hydrogen and carbon monoxide mixtures. Other considerations are the lifetimes of the electrocatalysts so that they may be considered for use in practical fuel cell power plants. At this time, most of the patent literature and scientific literature has been reviewed. The characteristics for anode electrocatalysts are considerably different from the characteristics of the cathode electrocatalysts, particularly when alloys or intermetallic compounds are being considered. That is to say, materials that have a high activity for hydrogen oxidation in the presence of catalyst poisons may be unstable or inactive for oxygen reduction.

The emphasis for phosphoric acid fuel cell electrocatalyst technology has been on platinum supported on a carbon for both the anode and the cathode electrocatalyst materials. Clearly, the emphasis for future electrocatalyst development must be on substituting for the platinum in the fuel cell system with equally active but lower cost materials.

A more detailed description of the electrocatalyst survey will be given in a latter report. At this time, however, we would like to show the phase diagrams of some of the most promising materials that have been developed for oxygen electrocatalysts (see P.N. Ross, Jr., U.S. DOE Contract W-7405-ENG-48, EPRI RP-1200-5, LBL-10799). The thrust of this work has been to develop noble metal-refractory metal intermetallics. The noble metals have been either platinum or palladium and the refractory metals have been from the carbide-forming refractory materials, principally Zr, Hf, V, Nb and Ta. The most interesting feature about the phase diagrams of the latter materials with both platinum and palladium is that a series of well-identified intermetallics are formed. The carbide-forming refractory metals occur with body-centered cubic structures, whereas the transition metals (nickel, palladium and platinum) have face-centered cubic structures. As such, the transition metals have a high electrocatalytic activity for both hydrogen oxidation and oxygen reduction by having a "close-packed" structure. Clearly, an intermetallic formed between the transition metals and the refractory metals should have a corresponding "close-packed" structure.

The most promising intermetallics are: ZrPt_4 , HfPt_4 , VPt_3 , NbPt_5 and TaPt_5 . Figure 13 shows the best phase diagrams that have been obtained to date for the nickel, palladium and platinum metals with the refractory metals. In this figure, the transition metal is plotted on the left hand side of the phase diagram and the refractory metal on the right hand side. The vertical dimension in the phase diagram is a temperature axis, going from 500-3500°C. Phases are marked as α (BCC), γ (FCC) and ϵ (HCP). The similarities and trends between the nickel, palladium and platinum phase diagrams with the same refractory metal become obvious. Of the intermetallics so far reported, the VPt_3 and TaPt_5 are the most promising, in that they appear to out-perform platinum for oxygen reduction in hot phosphoric acid (180°C), whilst at the same time they do not appear to corrode significantly. An unknown is the degree of demetallization (losing the refractory metal) at the particle surfaces.

For the anode, the most important consideration is that the electrocatalyst should be capable of oxidizing hydrogen in the presence of carbon monoxide rather than the oxidation of hydrogen in the absence of this poison. Alloying of platinum with rhodium (J. Electroanal. Chem., 1975, 59, 177-189) showed increased activities over that of platinum alone but during extended operation of the electrocatalyst, the rhodium (which is far more expensive than platinum) segregated to the insides of the alloy crystallites and the platinum to the surfaces. This had the effect of removing the most expensive component from beneficial operation. In an attempt to use a cheaper element to replace platinum, the platinum-ruthenium alloy combinations were examined (J. Electroanal. Chem., 1975, 63, 97-110). No great beneficial effects were identified. At various times, platinum-gold alloys have been examined as electrocatalysts but they are clearly inferior for hydrogen oxidation. At low temperatures, segregation occurs between a platinum-rich phase and a gold-rich phase, with the gold-rich phase at the electrocatalyst surface. Since gold is not active for the dissociation of hydrogen molecules, this electrocatalyst alloy combination is not satisfactory. On the other hand, substituting palladium for the platinum does produce an active electrocatalyst for hydrogen oxidation in the presence of carbon monoxide since palladium and gold form a continuous series of solid solutions (see Hansen Constitution of Binary Alloys, Second Edition, McGraw-Hill, P. 224). It is clear that sufficient palladium will be surface segregated on the palladium-gold alloy to confer electrocatalytic activity for hydrogen oxidation and yet carbon monoxide poisoning would not be overly significant. Fishman (U.S. Patent 3,510,355) produced palladium-gold

alloys as metal blacks and operated them in 85% H_3PO_4 at 75°C. Although the fuel only contained 0.1% carbon monoxide, he claimed that an active electrocatalyst was produced.

MATERIAL EDITED FROM THIS REPORT PENDING

NASA/DOE PATENT FINDINGS

35. Task 5 - Preparation of Platinum-Based Carbon-Supported Electrocatalysts. The purpose of this task is to prepare electrocatalysts that have been identified as promising candidates from the literature survey carried out in Task 4. It is intended that two platinum group metal (PGM) compounds shall be selected and three platinum group metal alloys. Selection of the compounds or the alloys is determined by the application of the material as an anode electrocatalyst or a cathode electrocatalyst. In addition, the electrocatalysts shall be cost effective with regard to providing an equivalent performance to a pure platinum catalyst but without having that

cost attributed to the platinum.

These electrocatalysts were bonded with PTFE and tested at 180°C in 100% phosphoric acid. In order to provide a rigorous evaluation of the catalytic activity it was decided that performance data would be obtained for hydrogen oxidation in the presence of 10% carbon monoxide and 30% carbon monoxide since these are the most difficult conditions for an anode electrocatalyst.

Results are shown in Figures 14 and 15. It can be seen that the alloys (Code EC 101) are very close to the performances of the good platinum electrodes, even though the PGM loading is 0.3 mg per cm² versus 0.5 mg per cm² for the platinum.

In order to chart the progress of our electrocatalyst development, we have constructed a plot of the electrocatalyst performance versus the dollar cost for the anode electrocatalyst material. This is shown in Figure 16. The dollar cost is normalized to the dollar cost of a platinum electrocatalyst. The performance criteria are those voltage losses for the electrode when operating at 200 mA per cm² and 180°C. Data are indicated in this figure for the 10% CO and the 30% CO gas mixtures. It can be seen that at this time we have lowered the anode electrocatalyst cost by over 50% while maintaining the electrocatalyst performance. Based on our projections for the new electrocatalyst materials, we think that it is possible to further halve this catalyst cost from the point that we have now achieved.

Appendix

Improvements in Test Equipment Operation

An operational problem exists in many laboratories when long term electrochemical experiments are carried out to obtain lifetime information on electrocatalysts. This problem comes about when the line power to the laboratory is either reduced, or a power outage occurs due to weather conditions. In the New England area, we had several power outages last winter which caused termination of some of our long term performance evaluations for electrocatalysts and carbon supports. The basic problem arises, not when the power goes off, but when the power is restored to the electrochemical equipment so that it comes on in an uncontrolled manner. Due to the operation of the Edelpol reference electrodes, which are electronically poised at the reversible hydrogen potential but without any passage of hydrogen (OSHA is decidedly unenthusiastic about bubbling hydrogen in a laboratory), power outages cause the Edelpols to relax to a platinum oxide potential, which is of the order of .8V vs hydrogen. Consequently, when power is restored and the potentiostats come on, the test electrodes are subjected to a very high potential. What is needed is an isolation circuit that senses a power outage and will block power to the potentiostats so that they can be reset in a controlled manner. Such a circuit has been designed and is shown in Figure 5. This circuit is described as a "power block" and is inserted between the line and the potentiostat controller. In the event that power is removed due to a switching transient, the "power block" circuit is designed not to activate, since a small switching transient will not effect the Edelpol references. In the event that a short (but longer than momentary) power outage occurs the "power block" circuit will still not activate unless the power outage is greater than 1.5 seconds. If the power outage is longer than 1.5 seconds, the circuit is activated and grabs the line power from the potentiostat. This means that the electrochemical controller can then be brought on line in a controlled manner without damage to the experimental electrodes. It provides operator control over the test system. We are contributing this circuitry since we believe that there may be many laboratories that have similar problems.

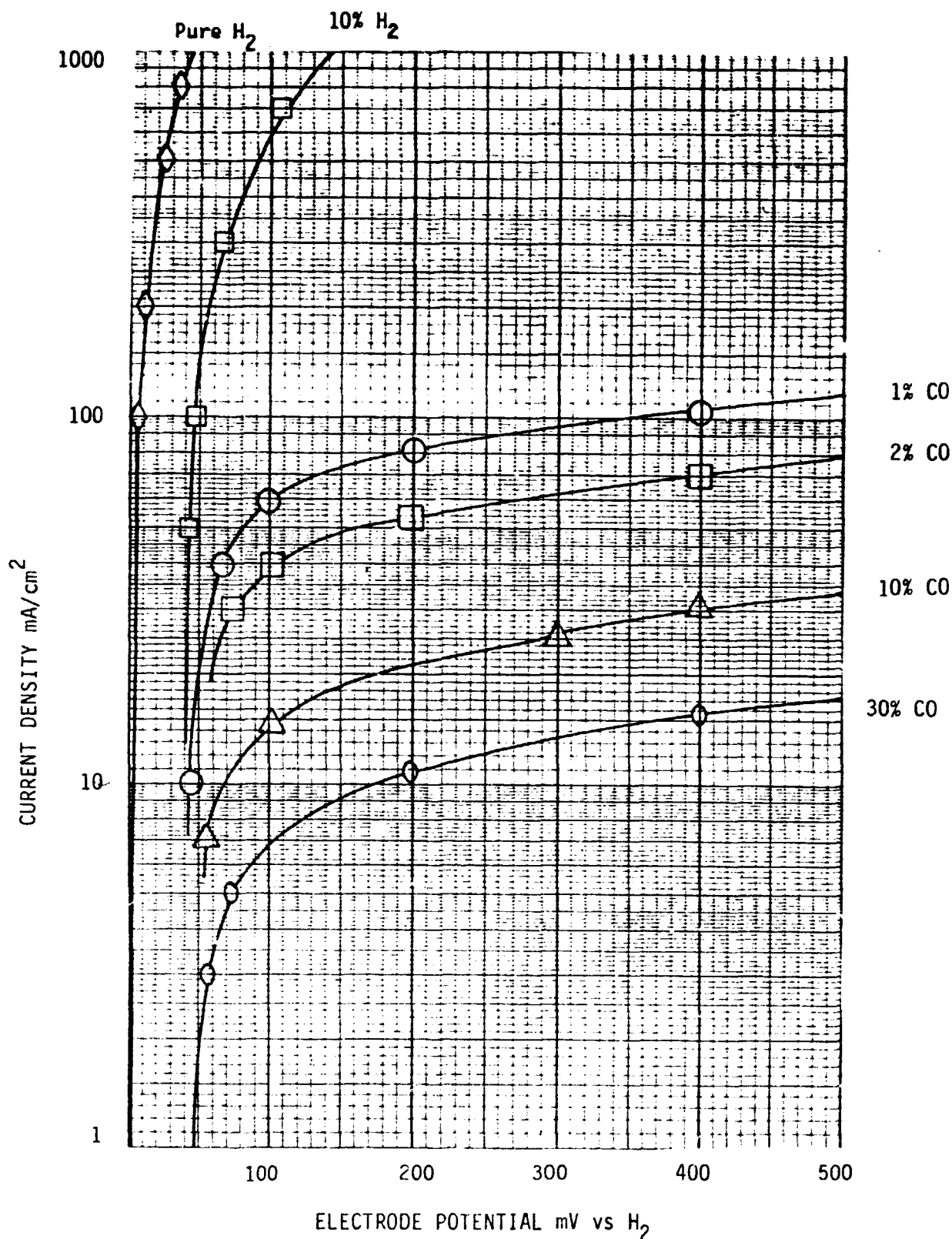


Figure 1. iR free polarization curves for hydrogen oxidation with various additions of CO to 10% H₂/N₂ fuel gas. Electrode contains 2½% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄.

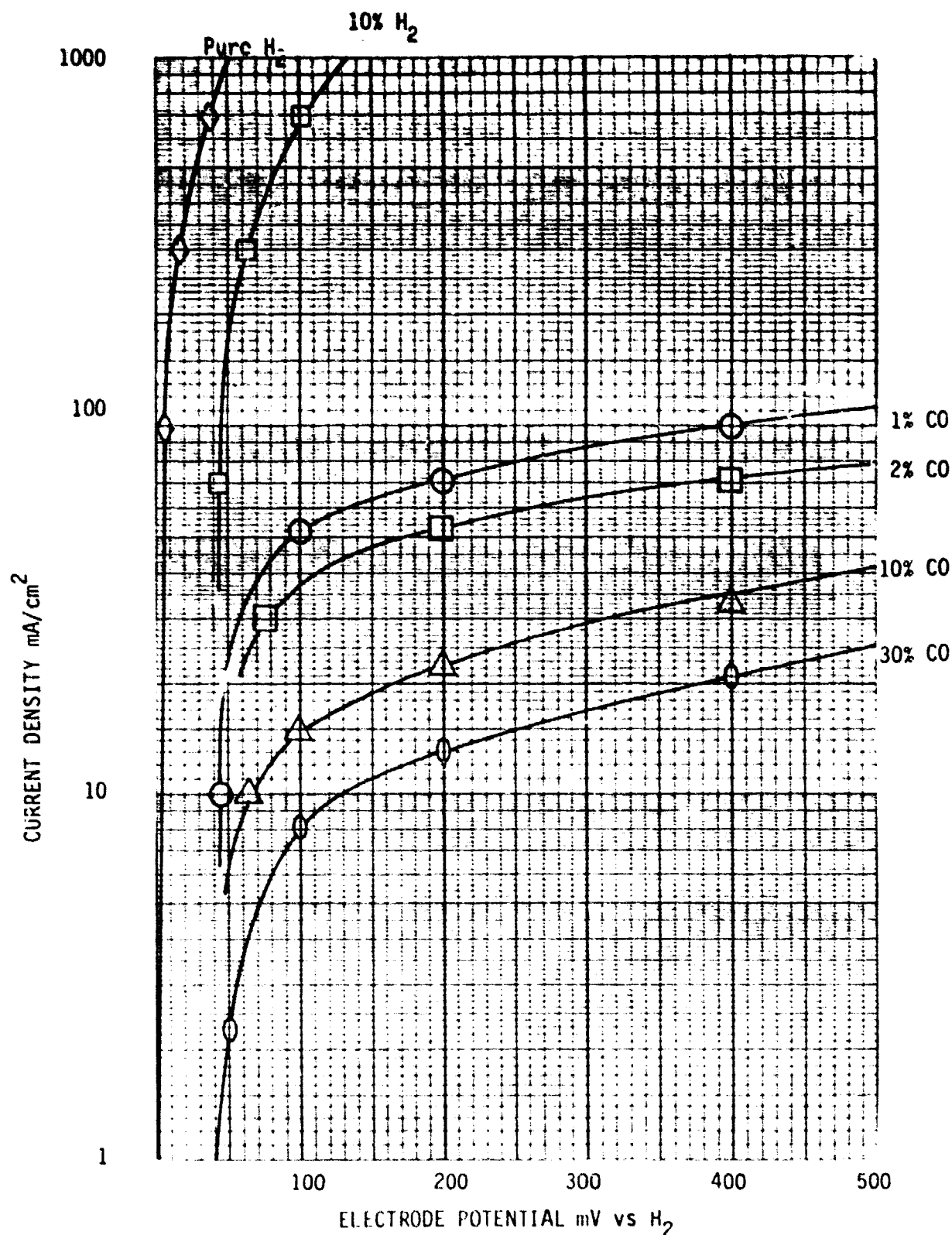


Figure 2. iR free polarization curves for hydrogen oxidation with various additions of CO to 10% H₂/N₂ fuel gas. Electrode contains 10% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄.

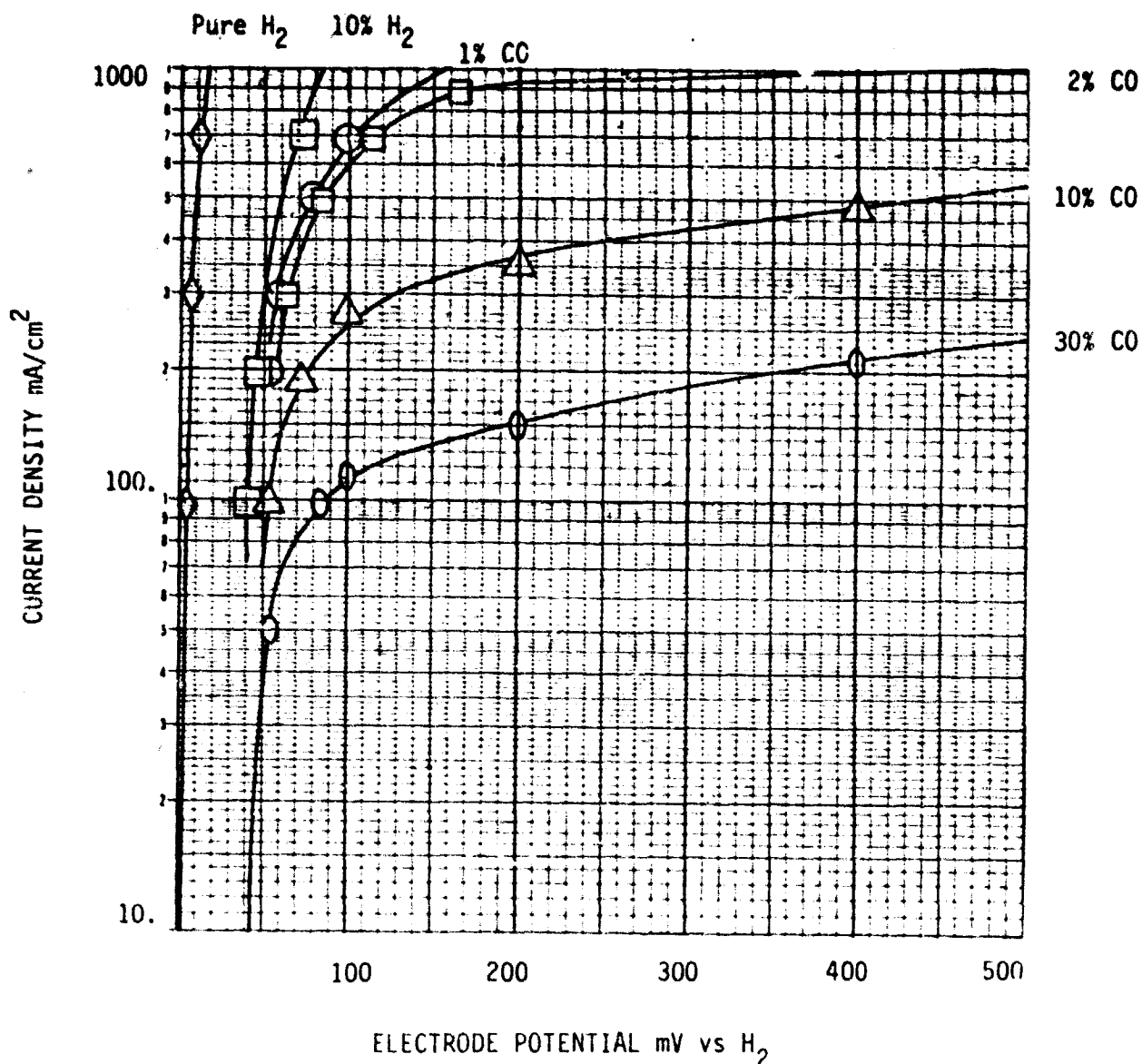


Figure 3. iR free polarization curves for hydrogen oxidation with various additions of CO to 10% H₂/N₂ fuel gas. Electrode contains 20% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄.

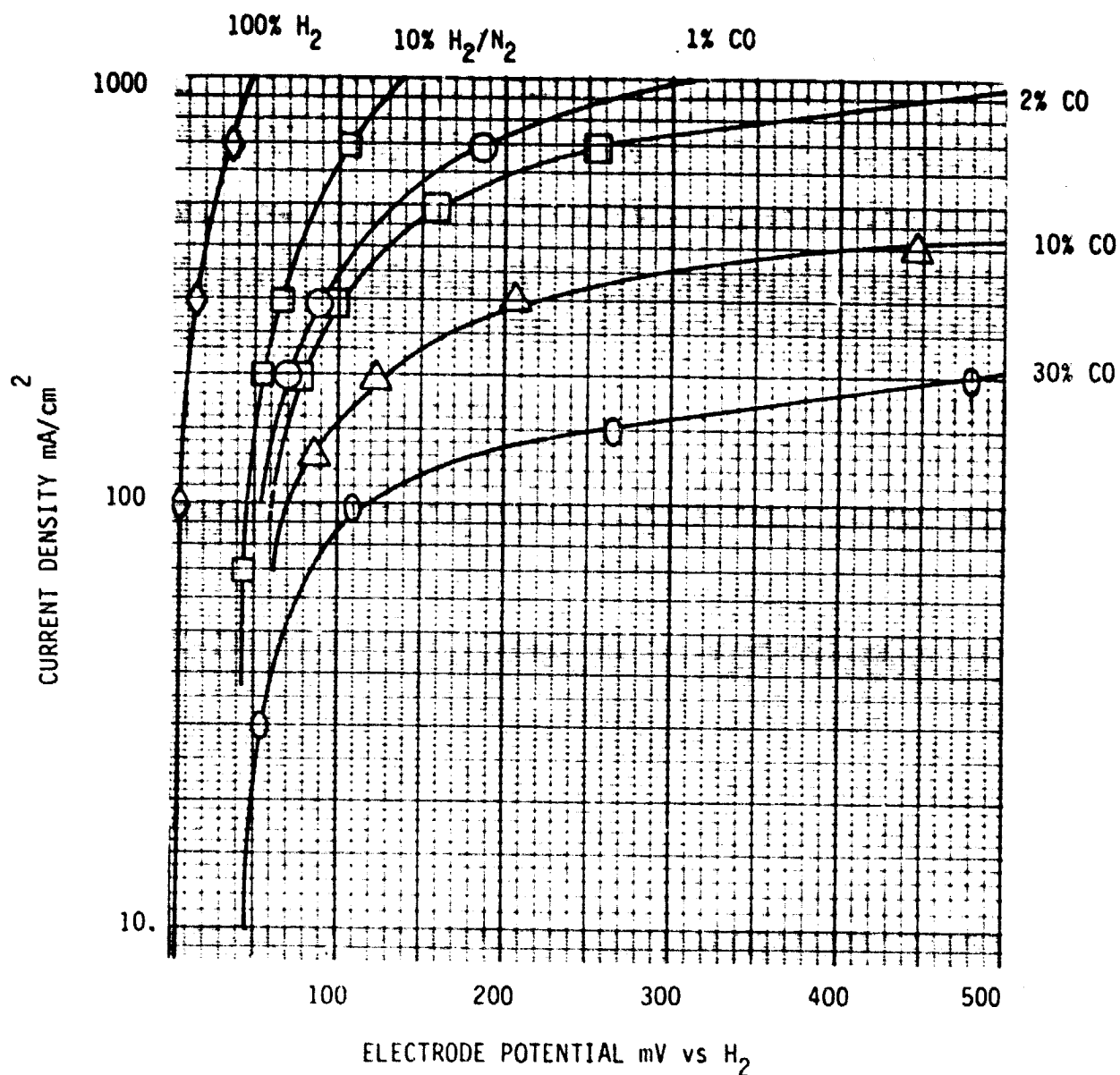


Figure 4. iR free polarization curves for hydrogen oxidation with various additions of CO to 10% H₂/N₂ fuel gas. Electrode contains 30% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄.

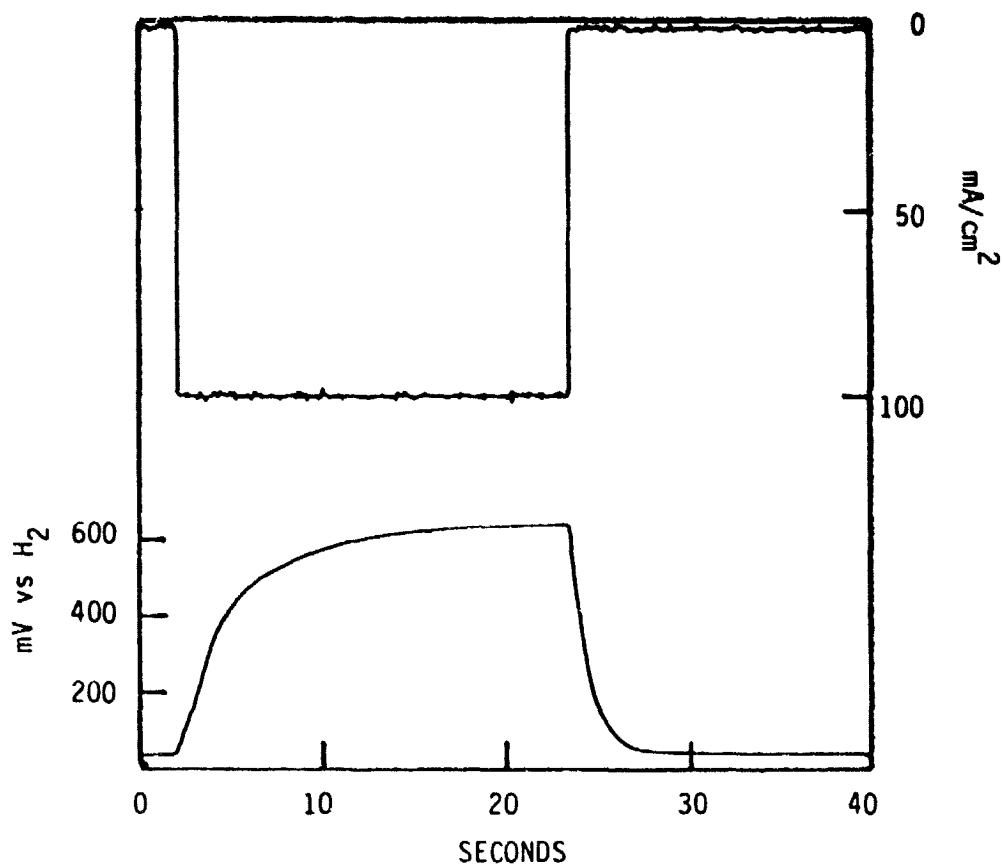


Figure 5. Response of an electrode containing 2½% PTFE to a galvanostatic transient (0 to 100mA). Upper trace is applied galvanostatic transient. Lower trace is potential response. Electrode contains 2½% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

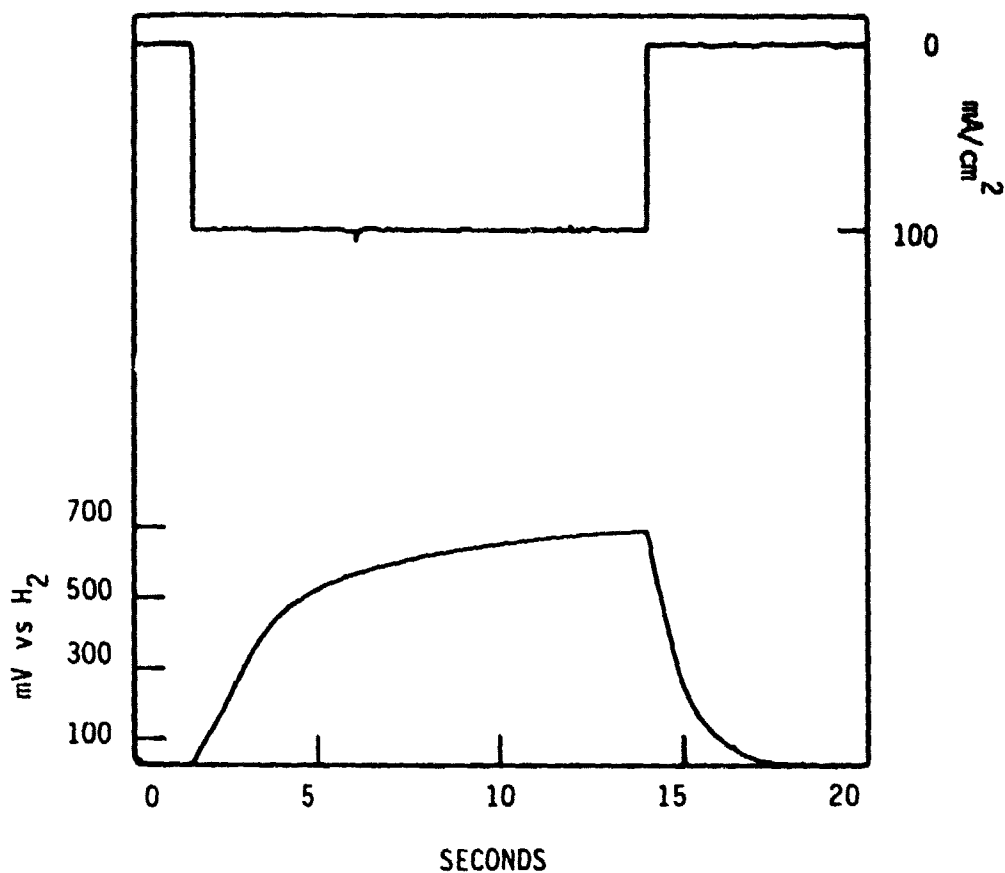


Figure 6. Response of an electrode containing 10% PTFE to a galvanostatic transient (0 to 100mA). Upper trace is applied galvanostatic transient. Lower trace is potential response. Electrode contains 10% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

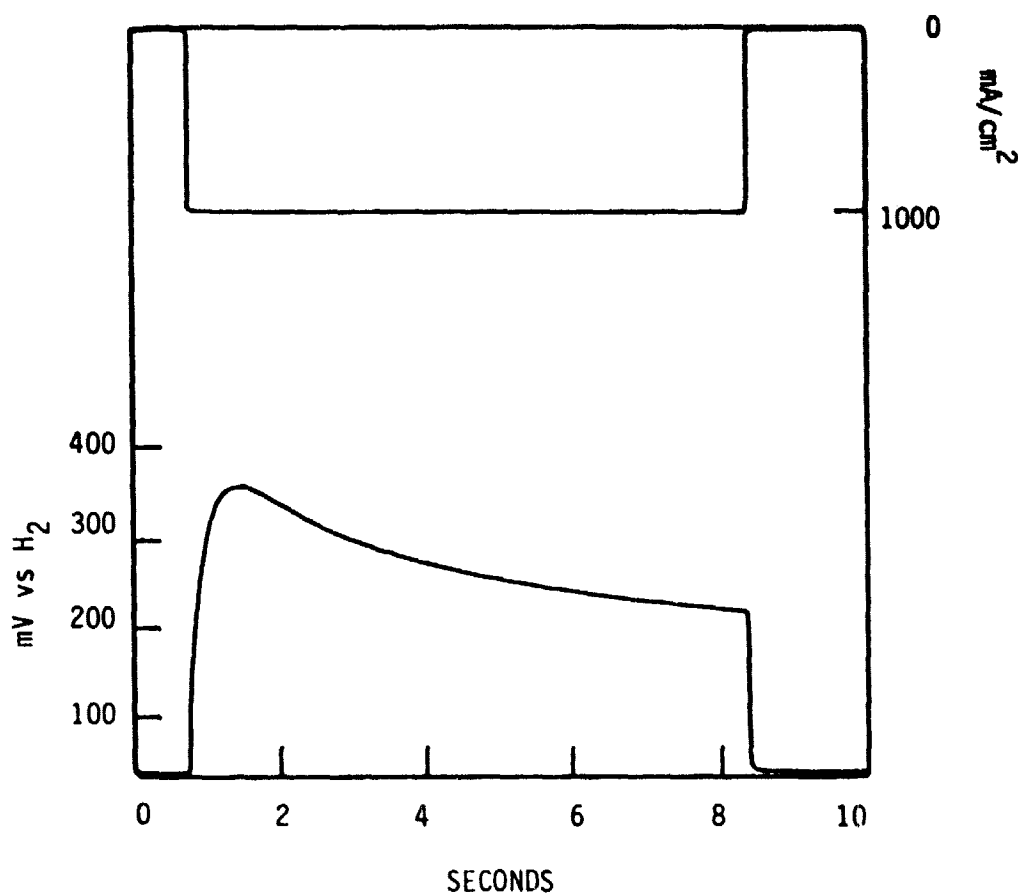


Figure 7. Response of an electrode containing 20% PTFE to a galvanostatic transient (0 to 1000mA). Upper trace is applied galvanostatic transient. Lower trace is potential response. Electrode contains 20% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

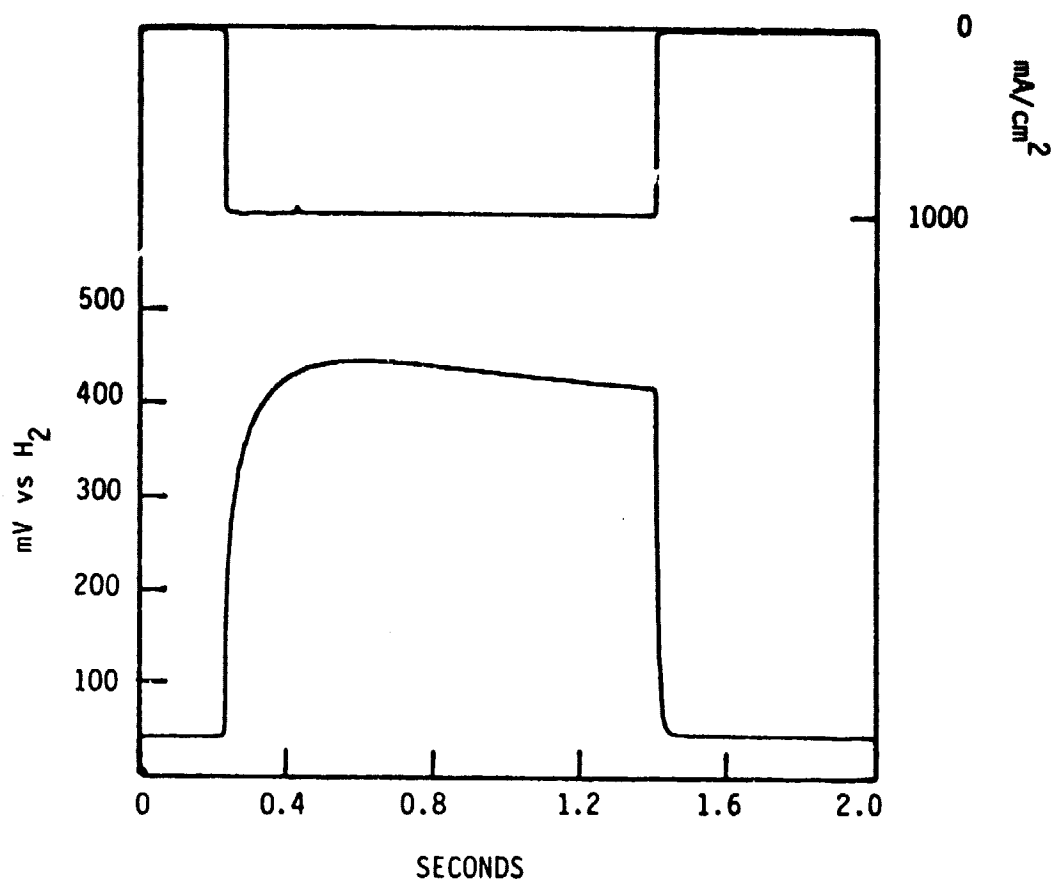


Figure 8. Response of an electrode containing 30% PTFE to a galvanostatic transient (0 to 1000mA). Upper trace is applied galvanostatic transient. Lower trace is potential response. Electrode contains 30% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

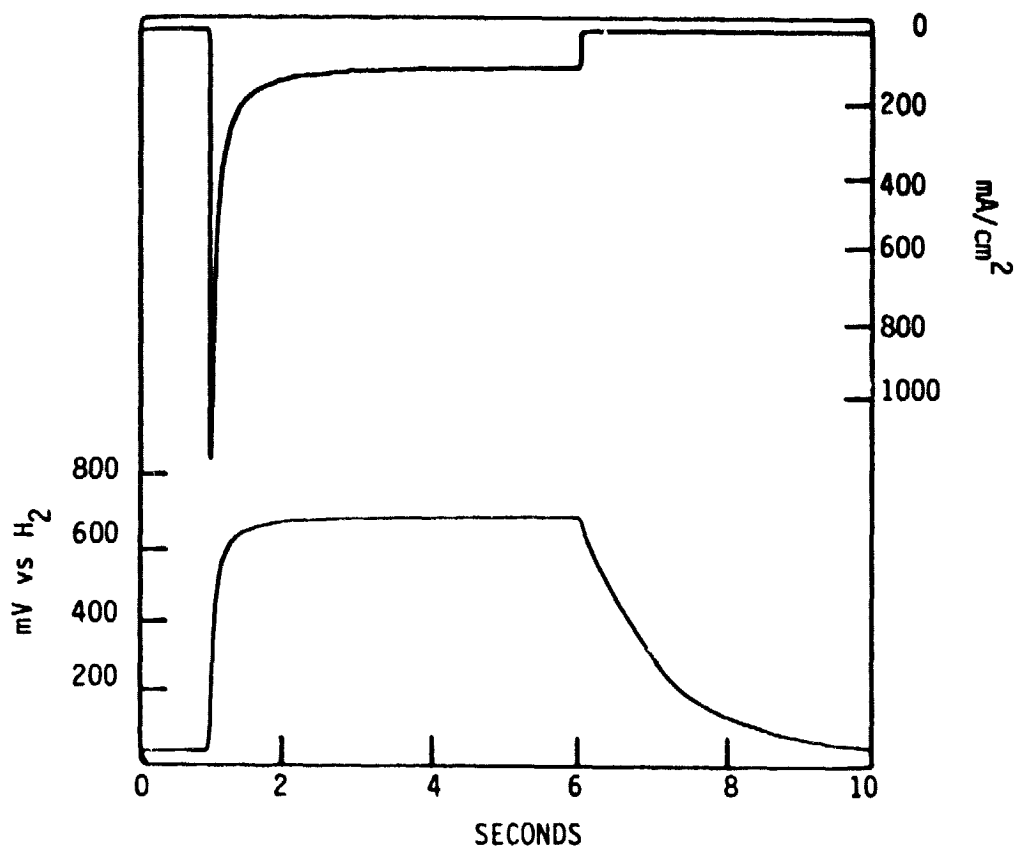


Figure 9. Response of an electrode containing 2½% PTFE to a potentiostatic transient (o.c. to 650 mV). Upper trace is current response. Lower trace is potential response. Electrode contains 2½% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

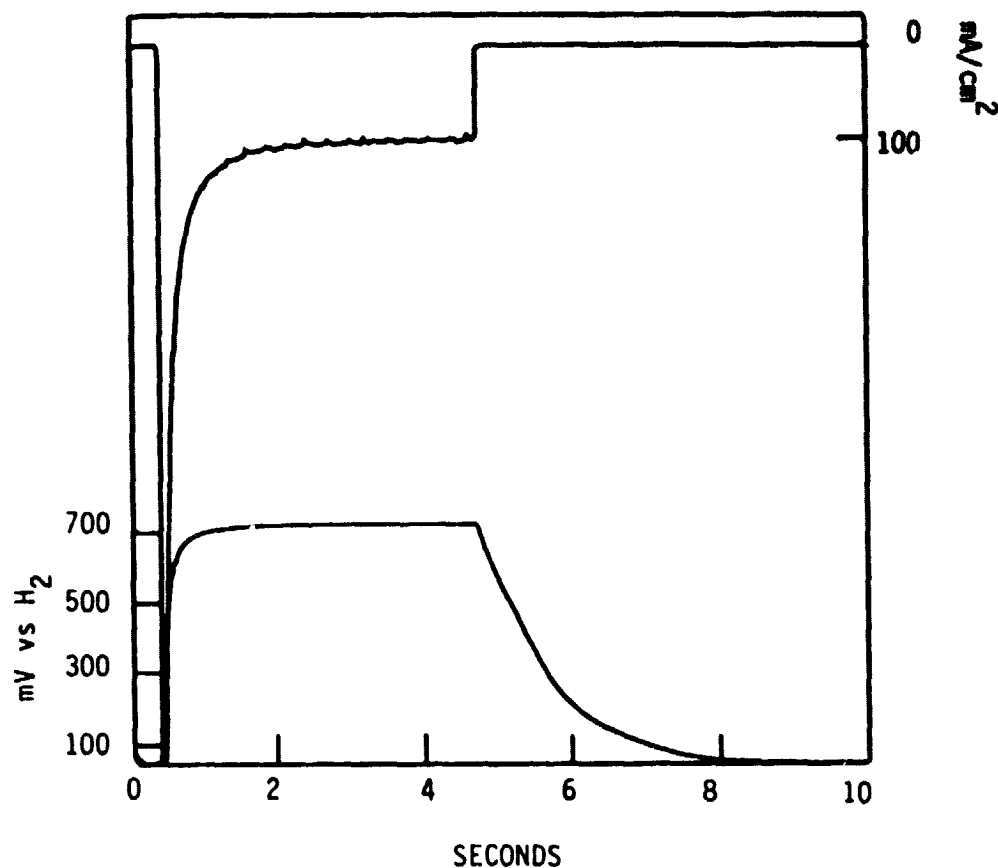


Figure 10. Response of an electrode containing 10% PTFE to a potentiostatic transient (o.c. to 700 mV). Upper trace is current response. Lower trace is potential response. Electrode contains 10% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

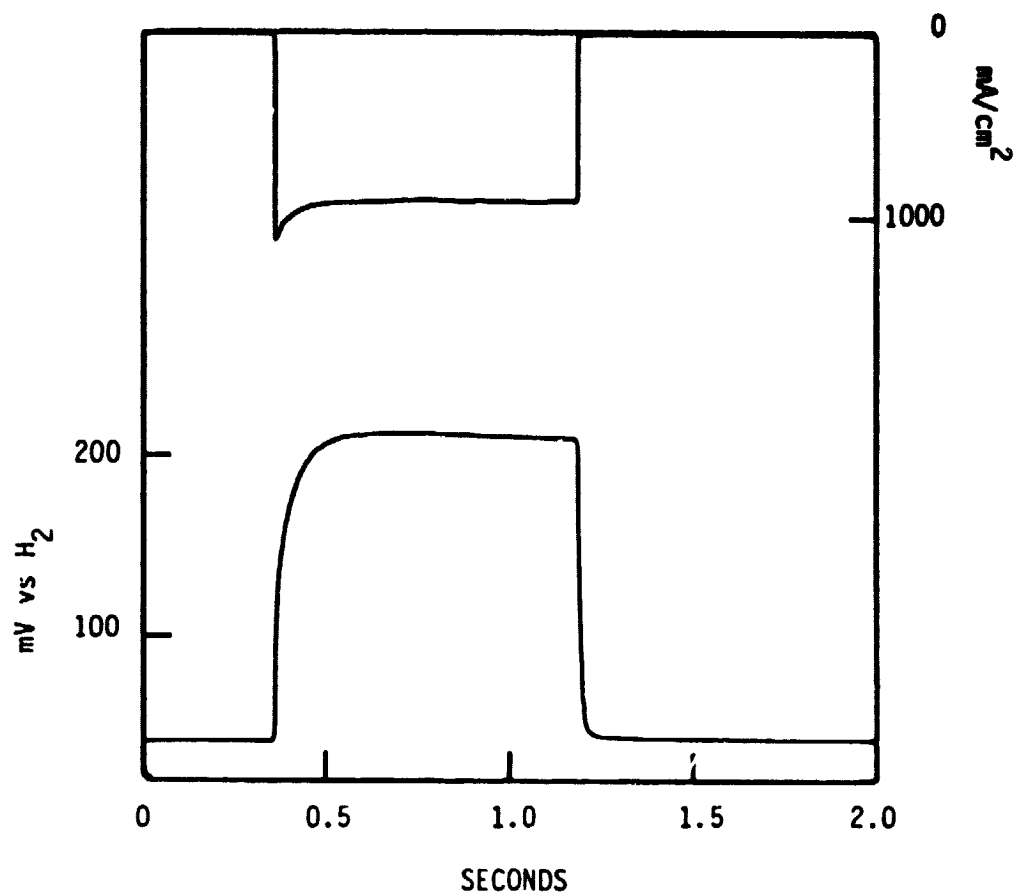


Figure 11. Response of an electrode containing 20% PTFE to a potentiostatic transient (o.c. to 200 mV). Upper trace is current response. Lower trace is potential response. Electrode contains 20% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

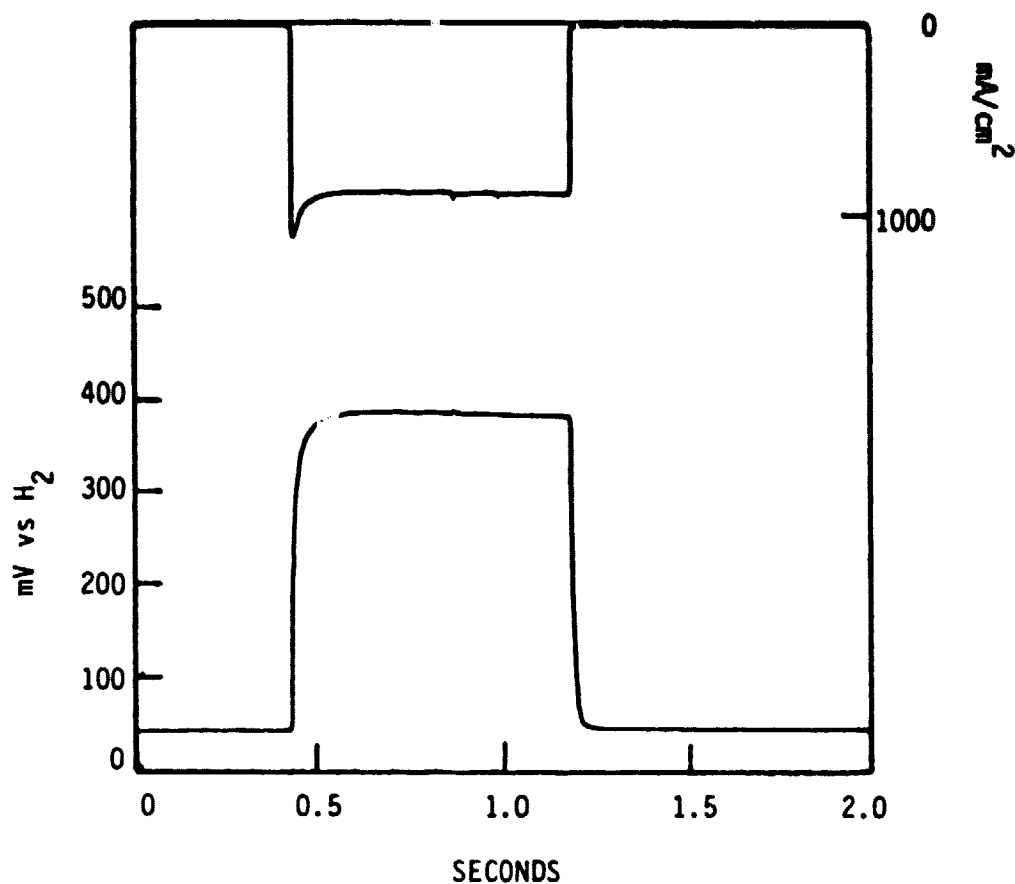


Figure 12. Response of an electrode containing 30% PTFE to a potentiostatic transient (o.c. to 380 mV). Upper trace is current response. Lower trace is potential response. Electrode contains 30% PTFE, 0.5 mg Pt/cm², was made from 10% Pt/Consel I and run at 180°C in 100% H₃PO₄, on 2% CO, 10% H₂, 88% N₂.

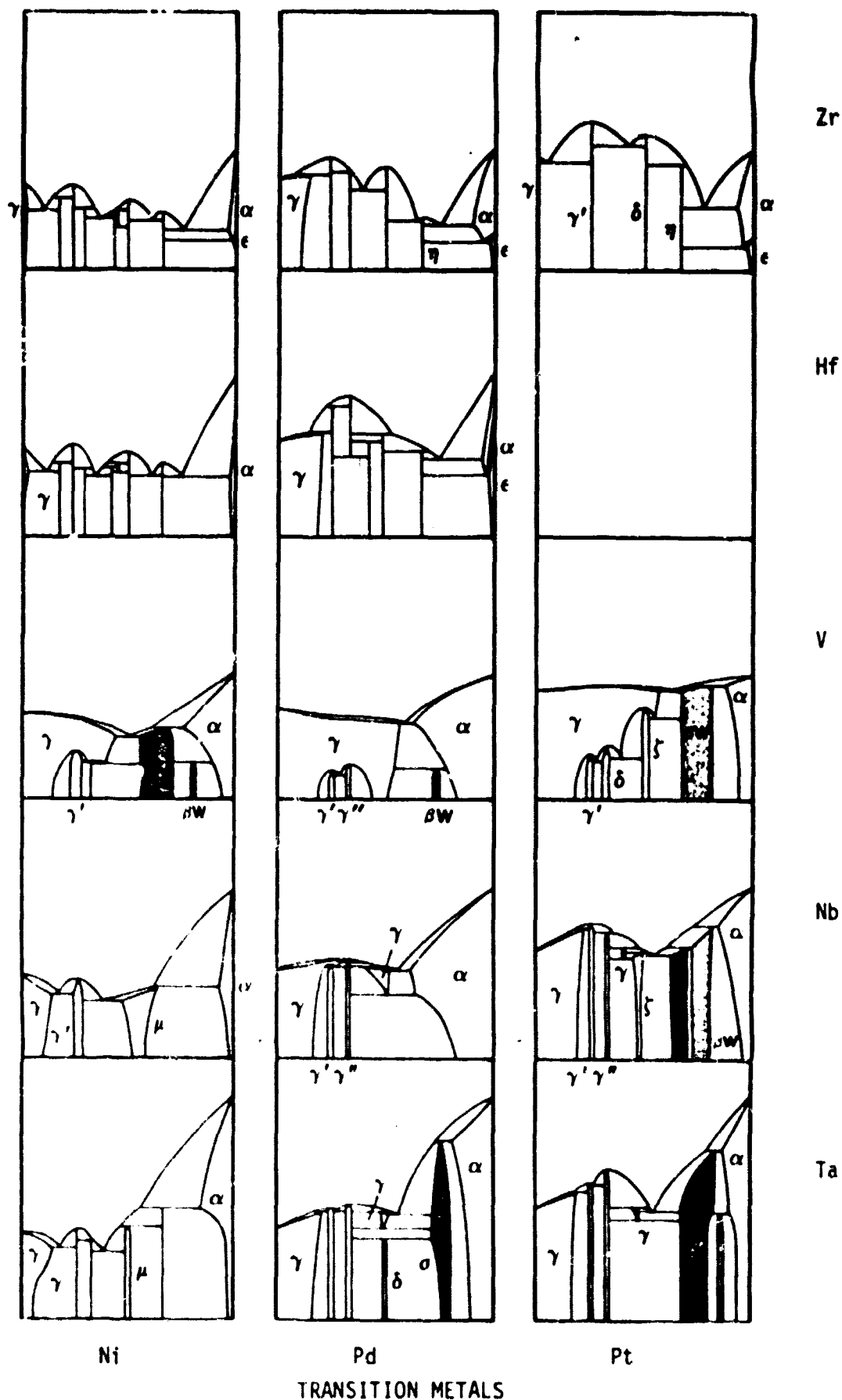


Figure 13. Phase Diagrams of Nickel, Palladium and Platinum Transition Metals with Refractory Metals

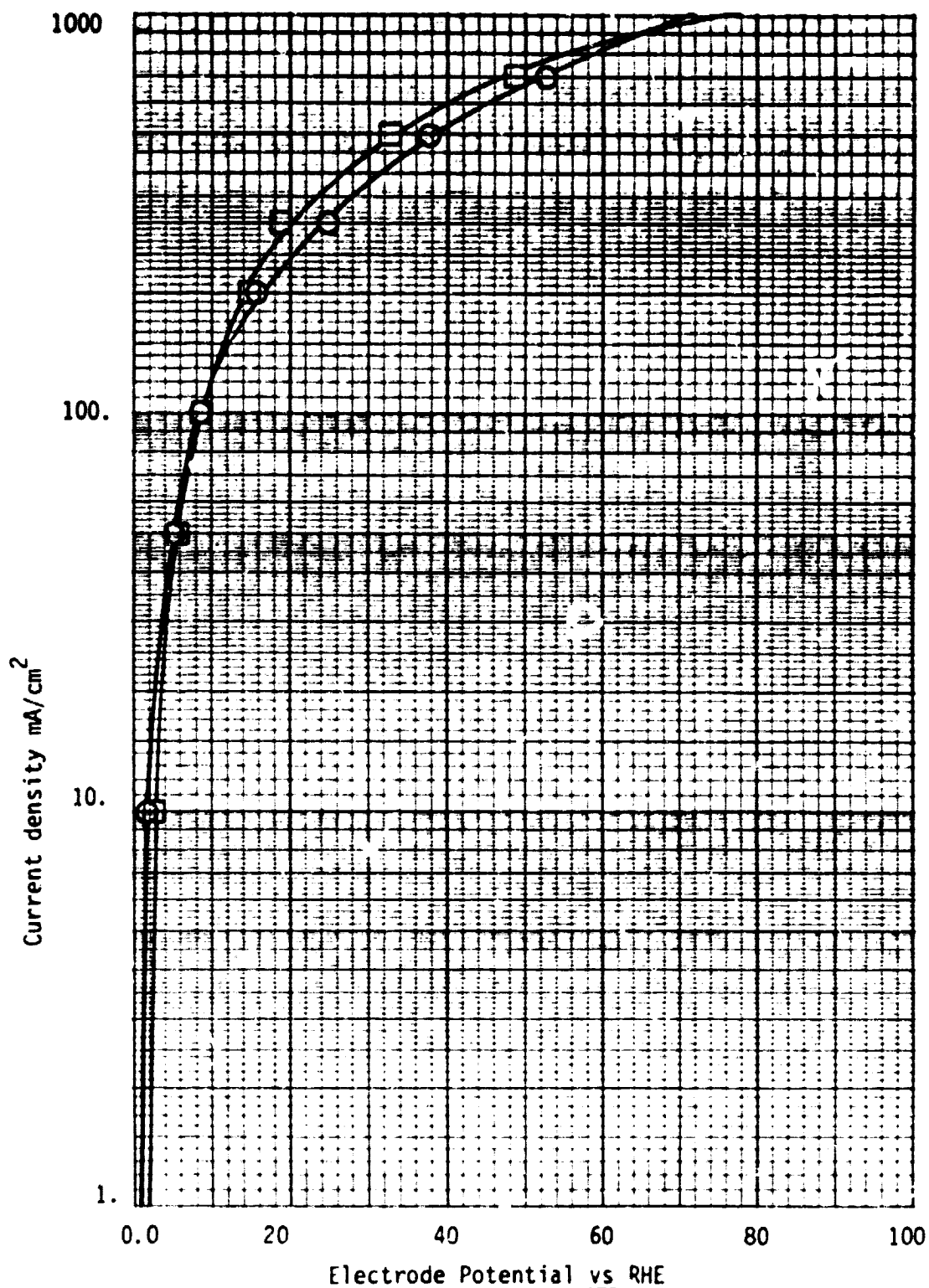


Figure 14.

Anode performances for 0.5mg Pt on Vulcan (squares) and 0.3mg PGM on carbon (circles). Nominal: 90% H_2 +10% CO . 180°C and 100% H_3PO_4 .

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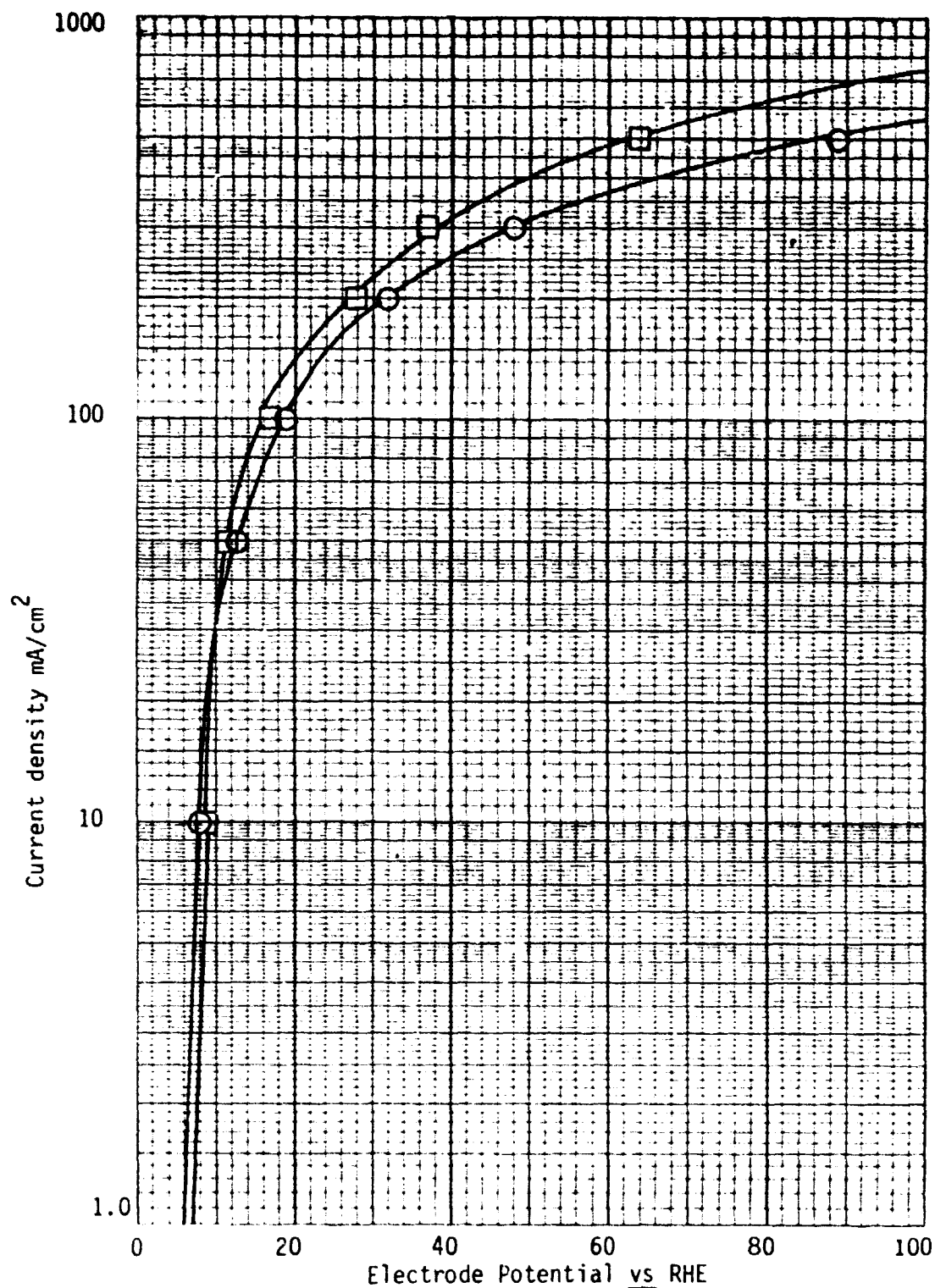


Figure 15.

Anode performances for 0.5mg Pt on Vulcan (squares) and 0.3mg PGM on carbon (circles). Nominal 70% H_2 +30% CO. 180°C and 100% H_3PO_4 .

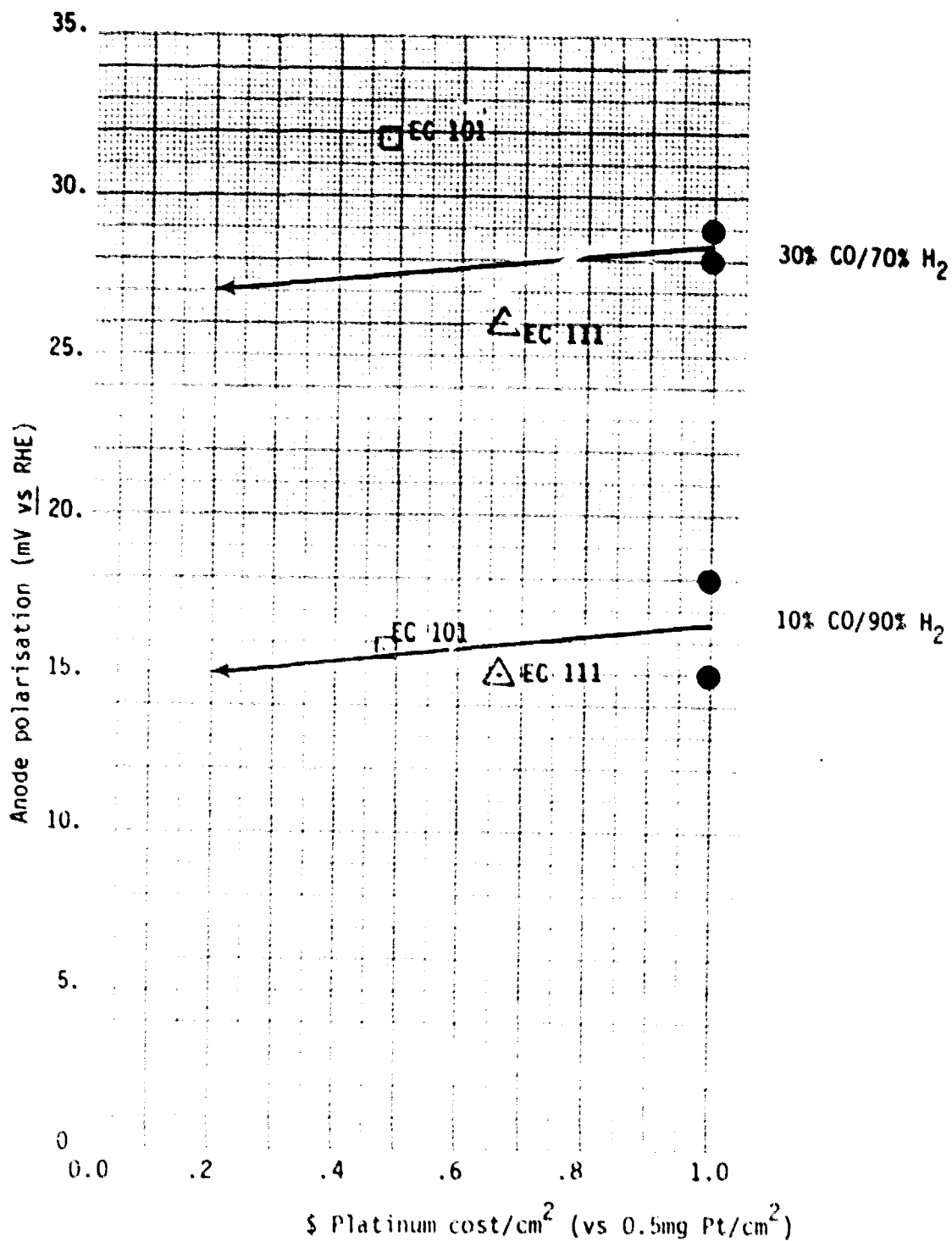
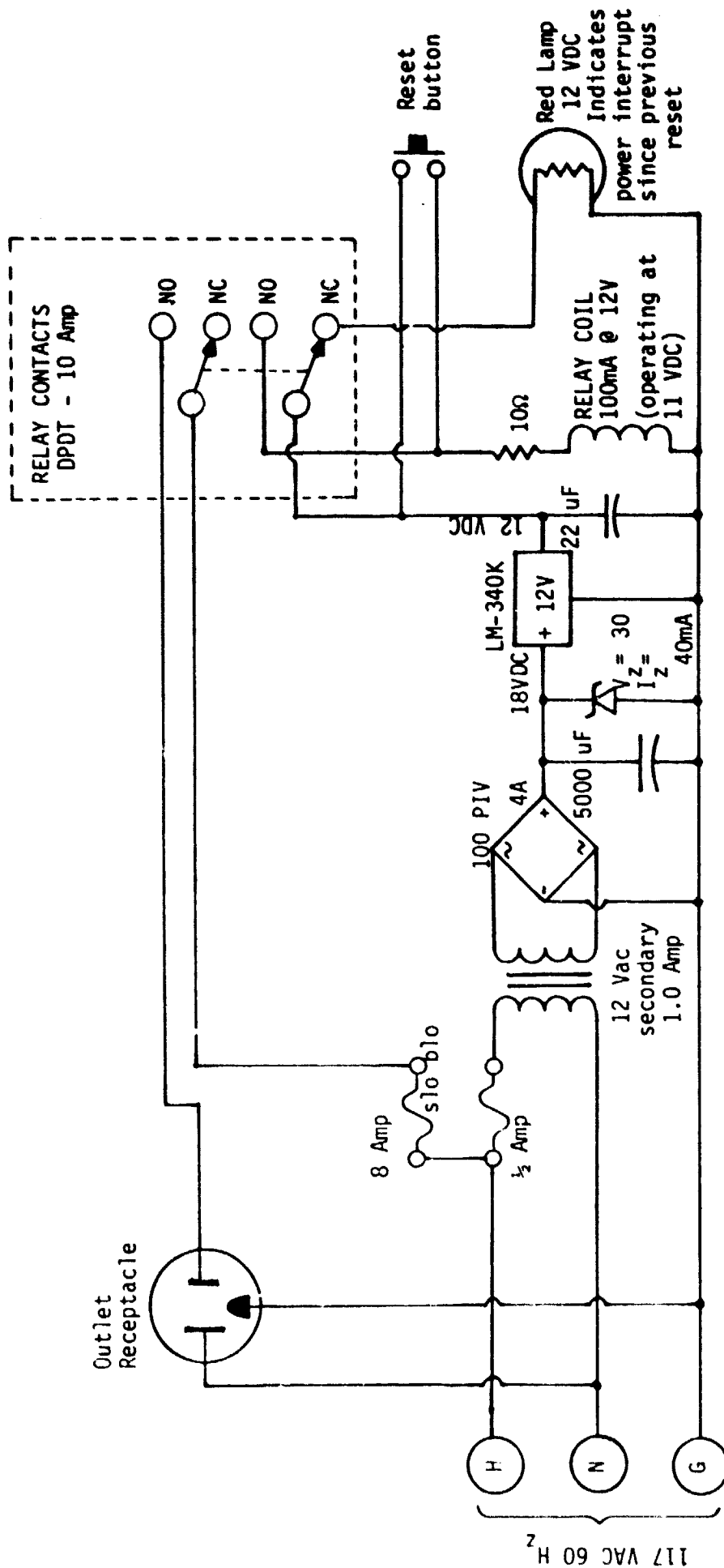


Figure 16. Comparisons of electrode performances for anode gas mixtures at 180°C in 100% H₃PO₄. Performance criterion at 200 mA/cm² electrode.⁴



ACTIVATES FOR POWER INTERRUPT OF 1.5 SECONDS OR LONGER

"POWER BLOCK" CIRCUIT

Figure 17.